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(54) Initiatory Explosives Comprising a Lead Salt of 3,5-Dinitro-catechol

(57) An initiatory explosive comprises the anhydrous normal lead (II) salt of 3,5 dinitro catechol (3,5-dinitro 1,2 dihydroxy benzene), which may be used alone or form part of an explosive composition mixed for example with oxidants, sensitizers etc. The explosive may be conveniently prepared by the reaction between dinitrocatechol and a suitable lead

compound, or by double decomposition, in aqueous media, and is suited for production in situ in an explosive device. It has a mechanical senstivity as good as conventional materials such as lead styphnate or resorcinate, but has a considerably lower electrostatic sensitivity, which leads to safer handling. The explosive and compositions containing it are suitable for use in most initiatory explosive devices such as percussion caps and stab sensitive detonators as a simple substitute for conventional materials.

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SPECIFICATION Initiatory Explosives

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This invention relates to metal salts of nitrated phenols which have explosive properties and may be used as initiatory explosives. Usually lead salts of nitrated phenols, especially poly-nitrated phenols such as 2,4-dinitroresorcinol, 4,6-dinitroresorcinol and 2,4,6-trinitroresorcinol (styphnic acid) have found extensive use as primary explosives.

In particular, both normal and monobasic lead styphnates are in general use in initiatory explosive compositions for example in priming compositions in percussion caps. In such applications the primary explosive is mixed with an oxidant such as a metal nitrate, chlorate or perchlorate, a sensitizer usually 10 tetrazene and usually one or more other ingredients such as lead dioxide, antimony sulphide, calcium silicide, aluminium and ground glass. Similarly lead 2,4-dinitroresorcinate is in general use in initiatory explosive compositions for example in composite detonators, mixed with an oxidant such as barium nitrate or potassium perchlorate, a sensitizer such as tetrazene and sometimes other additives such as aluminium or lead dioxide to provide an explosive composition sensitive to initiation by stabbing.

In addition to mechanical sensitiveness lead dinitroresorcinate and especially lead styphnate are also sensitive to electrostatic charges and stringent safety procedures are required in the manufacture 15 and handling of these explosives and derived compositions. Hazards in bulk preparation of these compositions may be considerably reduced if an explosive may be used possessing less electrostatic sensitiveness while maintaining the required mechanical sensitiveness. Alternatively the hazard may be 20 reduced by preparing the sensitive explosive ingredient in situ in the required composite mixture thus eliminating the need for prior manufacture in bulk of the sensitive explosive composition. Such a method for lead styphnate is claimed in Br. Pat. 1,479,569 but the efficacy of the method depends on the purity of the styphnic acid used.

According to this invention, an initiatory explosive comprises the anhydrous normal lead (II) salt 25 of 3.5-dinitrocatechol (3.5-dinitro-1,2-dihydroxybenzene) hereinafter termed lead DNC. The lead DNC may be used alone as a single component initiatory explosive but will normally form part of an explosive composition containing other ingredients of the types aforementioned, for example, in lead styphnate compositions. 3,5-dinitrocatechol (DNC) may be prepared by for example nitration of catechol as described in Chemische Berichte 26 (1893) p. 2183.

The lead (II) salt may then be prepared either by direct reaction of the DNC with a suitable lead 30 compound, such as the oxide, carbonate, formate or acetate, in the presence of moisture or, more conventionally, via a soluble DNC salt such as the sodium, potassium or magnesium salt by reaction with a soluble lead salt such as lead nitrate or lead acetate in aqueous solution.

According to a preferred aspect of the invention lead DNC is produced in situ in a detonator or 35 cap by filling the detonator or cap with a dry mixture comprising at least one precursor of the lead DNC and any other ingredients required in the final composition and subsequently adding an aqueous liquid to cause formation of the lead DNC. Thus the dry filling composition may contain DNC and lead oxide and the aqueous liquid is simply water. Alternatively the filling mixture may contain DNC but no lead compound when the aqueous liquid may be a solution or suspension of a lead salt, preferably lead 40 acetate. The detonator or cap is then heated to complete the reaction, if necessary, and to drive off the water and, in the case of using lead acetate, the acetic acid produced as a by product of the reaction.

In another aspect of the invention premixes of the above types may be prepared in much larger quantities with less hazard than for example lead styphnate compositions and converted in bulk by the aforescribed methods to the more sensitive explosive composition suitable for multiple filling of 45 detonators and caps. This method presents a simpler and less hazardous alternative to an existing type of process in which lead styphnate is prepared and handled in the wet state, then remotely dried and mixed with the other ingredients of the final composition. An advantage of the invention is that the normal drying cycle as used for wet lead styphnate is sufficient to complete the reaction between DNC and the lead compound, and dry the product.

The lead DNC produced by the processes described above shows similar mechanical 50 sensitiveness to lead styphnate (F of I (Rotter Test)=20) but considerably less electrostatic sensitiveness than lead styphnate both as a single explosive and in mixtures with other ingredients as mentioned previously.

Lead DNC is suitable for use in composite detonators using for example mixtures of lead 55 dinitroresorcinate, barium nitrate, tetrazene (L composition) or variants thereof as a stab sensitive increment, and in percussion caps using lead styphnate compositions such as VH2 (lead styphnate, barium nitrate, tetrazene, lead dioxide, antimony sulphide, calcium silicide) as a direct replacement for the lead DNR or the lead styphnate respectively. Lead DNC compositions of the above types are less violent than equivalent lead styphnate compositions but a more vigorous output may be obtained by 60 substituting the barium nitrate oxidant by potassium chlorate or potassium perchlorate. Thus a composition based on lead DNC, potassium perchlorate, tetrazene and, if necessary, a small amount of aluminium or other finely divided additive to aid mixing and filling operations, may be considered as a substitute for standard VH2 compositions.

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	The prepa example.	ration of lead DNC	using the met	hods outlined	l above will nov	w be describe	ed by way of	
	Example 1		of the	J. 48.		. • *5	e in the the terms	!
		d suspension of 2 g	3 5 DNC in 5	0 ml water e	• 2080		,-	
5	allowed to settle	e for 2 minutes, the wed by methylated	no surred for 3	iquid decent	efore cooling to	35°C. The p	product was	5 5
٠		0. 00 C. ,.	Yield: 10	0% Theoretic		•1		
10		read in Military to				· ·	1	10
	As a mode was made into a product was lea	el for in situ product a paste with 1.25 m d DNC.	tion of lead DN nl water and di	IC a mixture or ried on a hot	of 2 g 3.5 DNC plate at about !	and 2.23 g le 50°C. The res	ead monoxide sultant	: 10
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,				0% Theoretic				
20	Example 4				•		•	20
	As an alte	rnative to the in sit	u process of Ex	ample 1 a m	ixture of 2 g 3.	5 DNC and 3.	.79 g lead	
	acetate trinydra	te was moistened	with I—2 mily	vater, and dri	ied at about 50	°C to give lea	ad DNC.	
	Example 5	•	Yield: 10	00% Theoretic	cal		•	
25		ed solution of lead a	acetate tribydr	ata containin	a 2 70 a land a		•	
٠	added to 2 g 3.	5 DNC. The paste w	vas oried at ab	out 50°C to	yield lead DNC.	cetate trihydi	rate was	25
	Example 6		Yield: 10	0% Theoretic	al			
		of aqueous magne	sium 3 5-diniti	rocatechate c	Containing 20 s	// 2 E DNC	-4075.4	
30.	To 3 litres of aqueous magnesium 3,5-dinitrocatechate containing 30 g/l 3.5 DNC and 3.75 g/l magnesium oxide stirred at 65°C was added 0.541 aqueous lead acetate, containing 3.15 g/l lead							20
	acetate trinydrate, during 20 minutes. The mixture was stirred for a further 5 minutes at 65°C before						30	
		··· , -····· 5 -· · · · · · · · · ·	es. The mixtur	e was stirred	for a further 5	minutes at 6!	5°C before 😁	
	cooling to 35°.	The product was th	en treated as i	e was stirred n Example 1.	for a further 5	minutes at 6	5°C before	
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- 4. An initiatory explosive according to claim 3 also containing lead dioxide, antimony sulphide and calcium silicide.
- 5. An initiatory explosive according to claims 1 and 2 also containing potassium perchlorate and tetrazene.
 - 6. An explosive device containing an initiatory explosive according to any one of claims 1 to 5.
 - 7. An explosive device according to claim 6 which is mechanically fired.
- 8. A process for the production of an explosive device according to claim 5 or 6, wherein the said lead salt of dinitrocatechol is produced in situ in an explosive device.
- 9. A process according to claim 8 in which the said lead salt of dinitrocatechol is produced by 10 introducing a dry mixture comprising at least one precurso of said lead salt and any other ingredients required in the final composition into the explosive device, adding an aqueous liquid to form the lead salt, heating if necessary to complete the reaction and finally driving off remaining water and any
- 10. A process according to claim 9 in which the dry mixture contains lead oxide or a lead salt of a 15 volatile acid and dinitrocatechol, and the aqueous liquid comprises water.
 - 11. A process according to claim 9 in which the dry mixture contains dinitrocatechol, and the aqueous liquid comprises a solution of a lead salt of a volatile acid.
 - 12. A process according to claims 10 and 11 in which the lead salt is lead acetate.
 - 13. An explosive device prepared by a process according to any of claims 8 to 12.

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